Method and Device for Magnetron Sputtering

Applicant:

Fraunhofer-Gesellschaft zur Förderung der angewandten Forschung e.V.

The invention relates to a method and a device for magnetron sputtering. These technologies are used for depositing functional and finishing layers. Magnetron sputtering technologies are already used on a large scale in industrial production, e.g., for coating architectural glass.

Coating processes in which the coating is composed of several chemical elements are of particular technical importance. Titanium dioxide is one example. With coating processes of this type, the metallic component is often provided by sputtering a metallic target. The other layer component is introduced into the process chamber in a gaseous form. High coating rates and optimal layer quality can be achieved with these so-called reactive coating processes only when the process is operated in the area of unstable operating points. This so-called transition mode is characterized in that the reactive gas feed on the one hand is large enough to provide a sufficient amount of reactive gas for the layer deposit. On the other hand, however, the amount of reactive gas fed is so low that a contamination of the sputter target with reactive gas is avoided. Particularly with these unstable operating points, constant quality and reproducibility of the coating require the magnetron sputtering system to be operated with the aid of complex control loops.

According to the prior art, magnetron sputtering sources can be influenced through the electric power fed or the reactive gas flow. The necessary control signal can be obtained by measuring different parameters. Thus, for example, it is proposed in EP 1 232 293 B1 to use the harmonic content of the electric parameters of the discharge as control variable.

It is known from EP 0 795 623 A1 to determine the partial pressure of reactive gases with suitable probes. Thus, for example, the oxygen partial pressure measured with a lambda probe can be used as a control variable. It is known

from J. Affinito et al., J. Vac. Sci. Technol. A 2 (1984), p. 1275-1284, to control a magnetron sputtering source by measuring the plasma impedance. As a further possibility, the dissertation by J. Strümpfel, *Prozessstabilisierung beim reaktiven Hochratenzerstäuben mittels optischer Emissionsspektroskopie zur industriellen Herstellung von Indium-Zinn-Oxidschichten und Titandioxidschichten*, Chemnitz 1991, describes the measurement of the intensity of selected spectral lines of the plasma of the magnetron sputtering sources.

Furthermore, the deposition rate of a magnetron sputtering source that is operated in the unstable transition range is not absolutely known. The layer thicknesses of the layers produced on the substrate therefore have to be determined after deposition. Optical measurements such as photometry or ellipsometry are primarily used to this end.

The described expenditure in terms of equipment for each individual sputtering source of a coating system is necessary in order to ensure a constant layer quality, a constant layer structure and a constant layer thickness. This results on the one hand in high costs in acquiring and operating the coating system as well as in a high susceptibility to damage. This applies in particular to large in-line systems.

The coating of steel band substrates or architectural glass is carried out in continuous process on an industrial scale with in-line systems of this type. Such coating systems have a large number of magnetron sputtering sources. Twenty to approx. thirty sources are typical hereby, however, systems with up to sixty magnetron sputtering sources are also in use. What is critical for industrial production is thereby the interaction of all of these individual sputtering sources, whereby several sources often deposit one and the same material. Only through very great expenditure is it thereby possible for all the sources to supply identical results with respect to layer properties, coating rate and homogeneity.

An optical measurement of the layer properties after each sputtering source has proven to be extraordinarily difficult. In addition to high costs, in particular the

high susceptibility to damage of this overall system, which has a plurality of optical measuring systems, makes such methods inapplicable in practice.

The object of the invention is therefore to disclose a sputtering source which, without monitoring of the deposited layer and without complex control loops, makes it possible to deposit layers with defined properties and with defined coating rates. Furthermore, the object is to disclose a sputtering source that has a higher coating rate compared to the prior art for high-refractive materials such as titanium dioxide.

The object is attained through a magnetron coating system according to independent claim one and a method for depositing thin layers according to independent claim seven. Preferred embodiments are found in the respective dependent claims.

The magnetron coating system according to the invention comprises a first coating source, an auxiliary substrate, which is arranged between this first coating source and the area which is provided to receive the substrate to be coated, and a magnetron. Means are thereby provided for determining the area density of this auxiliary substrate, and the auxiliary substrate forms a cathode for the referenced magnetron.

To deposit a layer on a substrate accordingly first a layer is deposited with known deposit rate by means of the first coating source onto an auxiliary substrate. This auxiliary substrate now serves as a sputtering cathode for coating the substrate by means of the magnetron. Of course, not only the layer deposited on the auxiliary substrate can thereby be removed, but also the material of the auxiliary substrate itself. In this case both materials, optionally together with a component fed in a gaseous form, form the final layer on the substrate.

After the determination of the area density of the auxiliary substrate, the area density of the substrate can be determined from the mass balance of the auxiliary substrate. For example, a planar magnetron, a linear ion source, which sputters a target or implants xenon or krypton, a linear source that is based on

the principle of laser ablation or a linear evaporation source is suitable as the first coating source.

The auxiliary substrate is preferably embodied as a rotating, cylindrical body. Those areas that are facing the first coating source can thus be provided with a coating continuously, while at the same time those area elements that are facing the substrate are continuously available as sputtering cathode for coating the substrate. The auxiliary substrate is thus a component of a rod cathode magnetron. The cylindrical auxiliary substrate can be hollow in the interior area and thus tubular or embodied as a solid rod. The material sputtered off by the first magnetron can be continuously transported to the substrate and deposited there through the rotation of the auxiliary substrate.

In a particularly advantageous embodiment, the first coating source is a planar magnetron. This first magnetron is thereby operated in a pure inert atmosphere. The coating rate can thus be determined absolutely from the known sputtering rate and from the electric discharge parameters.

If now the second magnetron is operated with reactive gas or a mixture of inert and reactive gas, the coating rate of the substrate can be only inadequately quantified due to the constantly changing reactive gas partial pressure at this point. After the determination of the area density on the auxiliary substrate, however, the coating rate of the substrate can be determined absolutely from the mass balance of the auxiliary substrate.

From case to case the first coating source can be arranged in a shield in order to prevent the penetration of reactive gas components which would contaminate the coating source.

Advantageously the area density of the auxiliary substrate is determined by means of x-ray fluorescence. Overall in this manner the coating rate of the substrate can be determined with an error of less than 0.1%.

Argon in particular is suitable as inert gas for operating the first magnetron. This is available without major technical effort and at low cost. Moreover, argon as

an inert gas has a high ionization potential and remains inert even at high temperatures. Nitrogen and/or oxygen and/or methane in particular are suitable as a reactive gas. Thus in combination with a metallic sputter target nitrides, oxides or carbides can be deposited as a thin layer on the substrate.

In particular a metal layer of less than 100 nanometers, particularly preferably a layer of less than 10 nanometers thick, deposited on the auxiliary substrate is suitable as a metallic target. It is known from S. Berg, J. Vac. Sci. Technol. A 10 (1992), p. 1592-1596, that the sputtering rate of materials with implanted heavy atoms is clearly higher compared to the sputtering rate of the pure material. Thus with the magnetron coating system according to the invention high deposition rates can be achieved even with operating conditions outside the transition mode. For materials with a high refractive index, such as, e.g., titanium dioxide, the device according to the invention allows the coating rate to increase by more than 50%.

A particularly high increase in the coating rate results accordingly when the metal layer deposited by means of the first magnetron has a higher mass number than the average mass number of the material of the auxiliary substrate. Thus, for example, the sputtering rate of a 2 nm thick layer of tungsten on an auxiliary substrate of aluminum is greater by up to a factor of 3 than the sputtering rate of a homogenous tungsten target.

As is known from the prior art, the second magnetron can be operated as an individual magnetron with DC voltage or with pulsed DC voltage. Advantageously, however, the device according to the invention is operated as a double magnetron with an AC voltage of approx. 10 kHz to approx. 100 kHz. Operation at a frequency of 40 kHz is particularly advantageous. With operation as a double magnetron, two of the arrangements shown in Fig. 1 are connected to the poles of an AC source. Each auxiliary substrate is thus alternately switched as anode and cathode. An effective unification of the surfaces of the auxiliary substrates occurs through the alternating electron bombardment of the auxiliary substrates. This increases the process stability as desired.

Furthermore, the operation of at least two auxiliary substrates as a double magnetron leads to a greater plasma density and thus to improved layer properties as desired.

The magnetron coating system according to the invention thus makes it possible for the first time to monitor the achievement of a predetermined layer thickness in a simple manner with the aid of the integral measuring technique. With the aid of this technology, even large in-line sputtering systems with a plurality of coating stations can be realized, which were not manageable with the control methods and optical diagnosis systems hitherto available.

The invention is described below by way of example on the basis of one Fig.

Fig. 1 shows the diagrammatic structure of a magnetron coating module according to the present invention.

In the center the Fig. shows a cylindrical auxiliary substrate 2 that rotates about its longitudinal axis. The substrate 1 to be coated is arranged below the cylindrical auxiliary substrate. This substrate can be, e.g., architectural glass. The substrate 1 is moved through under the coating system. Plasma is ignited through a voltage applied to the auxiliary substrate 2, in the area 3 between the auxiliary substrate 2 and the substrate 1. The auxiliary substrate thus forms a rod cathode from which material is sputtered off which coats the substrate 1 switched as an anode. A mixture of inert and reactive gas is located in the area 3, which permits the deposit of a multi-component layer. A planar magnetron 5 in a shield 4 is located on the opposite side of the auxiliary substrate 2. In this case, the auxiliary substrate 2 is switched as an anode that is coated with material of the planar sputtering cathode 5 in the plasma area 7. The gas phase in area 7 contains exclusively inert gas so that the deposition rate in area 7 can be determined from the known sputtering rates and the electric parameters. The coating rate on the substrate 1 results from the mass balance on the auxiliary substrate 2. In addition to the known coating rate in the area 7, the area density after the sputtering process in area 3 is required to this end as well. To this end a device for determining the x-ray fluorescence 6 is located behind the plasma

zone 3. The device 6 thereby contains an x-ray source for irradiating the auxiliary substrate 2 and a photodetector for determining the x-ray radiation reflected from the auxiliary substrate.